L6: Entry 3 of 9 File: USPT Nov 4, 1997

DOCUMENT-IDENTIFIER: US 5683916 A
TITLE: Membrane affinity apparatus and purification methods related thereto

Brief Summary Paragraph Right (8):
The "ideal" column geometry would have an infinitely short bed height (to minimize operating pressures and maximize operating pressures and maximize throughput) and an infinite width (to maximize ligand loading and binding capacity). In reality, a substantially isotropic microporous hollow fiber membrane configuration approaches this ideal quite closely, with "bed heights" in the 300-micron range and large internal surface areas.

Brief Summary Paragraph Right (23):
U.S. Pat. No. 4,693,985 describes flat sheet polyamide membranes for affinity applications. These are microporous, skinless membranes with pore diameter of <0.1 to >0.45 .mu.m. They represent a major improvement over UF flat sheet membranes, but are still limited by being difficult to configure in a device (as they are not self supporting). Also, high surface area/low dead volume devices become increasingly difficult to design. In U.S. Pat. No. 4,693,985 pleated filter is described as the best available configuration for packaging flat sheet membrane in a high-surface-area configuration.

Brief Summary Paragraph Right (33):
A series of U.S. Pat. Nos. 4,473,474, 4,473,475, and 4,673,504, describes a method for the charge modification of a hydrophilic wettable membrane surface which utilizes crosslinking agents to form a covalent bond with the "hydroxyl, carboxyl, and primary and secondary amines, which are on the hydrophilic microporous membrane and the cationic charge modifying agent." Although these patents state that a covalent bond may form between amino and carboxyl groups on the surface of the preferred nylon 66 (a polyhexamethylene adipamide) membrane and an epoxy group of the crosslinking agent, they fail to disclose the source and origin of these functional groups and seem to suggest that hydroxyl, carboxyl, and amino groups are simply present on all hydrophilic surfaces including the nylon 66. In fact, polyamides cannot contain hydroxyl functional groups. All three patents expressly state that such hydrophilicity is a necessary element of that invention and the most recently issued patent states, again expressly, that hydrophobic polymer membranes are not amenable to charge modification by the methods of that invention. U.S. Pat. Nos. 4,711,793 and 4,708,803 issued to Ostreicher et al. relate to the same subject matter.

Brief Summary Paragraph Right (34):
U.S. Patents recently issued to Barnes et al. (U.S. Pat. Nos. 4,743,418 and 4,737,291) and European Patent Application 0 066 814 address a process for using 1,4-butanediol diglycidylether, specifically as a crosslinking agent for modifying the charge of a microporous nylon membrane. Again, these references fail to appreciate or teach the origin and nature of the "hydrophilic" functional groups on the membrane surface.

Brief Summary Paragraph Right (37):
In the specialized area of membrane art, the current methods for producing microporous membranes generally result in skinned anisotropic structures characterized by wide variations in pore sizes from the outer to the inner portions of the membrane. In particular, the production of isotropic hollow fiber membranes has been hampered by prevailing biases in the art and by existing extrusion methods, over and above the general manufacturing techniques.

Brief Summary Paragraph Right (41):
Traditionally, workers in the art have to take into account the pore size range of

interest in selecting the membrane polymer. It is believed that certain polymers are more readily processed to make membranes in certain pore-size ranges than others (See, Kesting, Syn. Polym. Memb., supra). Workers in the field such as Strathmann, et al., Desalination 1977, 21, 241-255 and Desalination 1975, 16, 179-203; Tanny, et al., J. Appl. Polym. Sci. 1974, 18, 2149-2163; Koenhen, D. M., et al., J. Appl. Polym. Sci. 1977, 21, 199-215; Broens, L., et al., Desalination 1977, 22, 205-219; Altena, F. W. and Smolders, C. A., J. Polym. Sci.: Polymer Symposium 1981, 69, 1-10; Broens, L., et al., Desalination 1980, 32, 33-45; Bokhorst, H. et al., Desalination 1981, 38, 349-360; Wijmans, J. G., et al., J. Memb. Sci. 1983, 14, 263-274; and Kesting have headed efforts toward a greater understanding of the mechanism of membrane formation and the ways of manipulating structural properties. It has generally been accepted in the field of membrane processing that many key manufacturing parameters have to be changed and tediously reoptimized in going from a flat sheet formulation to a hollow fiber product. Progress, has thus been slow, particularly with respect to the production of isotropic microporous hollow fiber membranes.

Brief Summary Paragraph Right (49):
In contrast to the nonsolvent induced liquid/solid phase separation for preparing essentially anisotropic microporous membranes, Castro (U.S. Pat. No. 4,247,498) has exploited thermal phase inversion (i.e., liquid/liquid phase separation brought about by temperature changes) in the preparation of isotropic microporous membranes. Thermal phase inversion, as it is currently practiced, requires a polymer melt and a compatible liquid to give a homogeneous solution in which the polymer is solubilized in the poor solvent. Subsequent cooling of these melts results in the precipitation of the polymer. The structure is thus "frozen" by the cooling process.

Brief Summary Paragraph Right (51):
Present spinnerette assemblies for hollow fiber manufacturing, are wholly inadequate and inflexible for the production of substantially isotropic microporous membranes. The extrusion dies currently in use do not provide the degree of control over the pore structure and pore-size distribution of the resulting microporous hollow fibers that one would wish to have. Typical tube-in-orifice spinneretres are described in U.S. Pat. Nos. 4,198,363 (Noel, G. et al.) and 4,229,154 (Chaban and Hawkins); in Borneman, Z. et al. Proceedings, 4th British Oxygen Company Conference, September 1986, p. 145-157; and in Aptel, P. et al. J. Memb. Sci. 1985, 22, 199-215. Spinnerette face plate configurations are further disclosed in an article by Cabasso in "Hollow Fiber Membranes," Encyclopedia of Chem. Tech., 3rd Edition, Vol. 12, p. 499, Kirth-Othmer, Eds.

Brief Summary Paragraph Right (58):
The apparatus of this invention is a cross flow, hollow fiber affinity membrane system for separation of high value products, such as therapeutic proteins. The central feature of the system is a substantially isostropic microporous hollow fiber membrane designed so as to optimize loading capacity and low dead volume while achieving high mass transfer rates. A large scale system having one 600 ml module is designed to process up to 10,000 liters of cell culture harvest per week. Multiple affinity systems or modules can be run in parallel in order to process even larger quantities of feed material.

Brief Summary Paragraph Right (63):
The apparatus is designed to meet criteria such as high volumetric throughput, high reliability, ease of scale-up, high selectivity and high product yield. The high volumetric throughput is accomplished by high filtrate flow rates enabled by the unique low pressure drop characteristic of the membrane process, but more particularly, by the substantially isotropic, micron pore size, microporous hollow fiber membrane of this invention.

Brief Summary Paragraph Right (75):
The present invention also describes the unique characteristics of a four-component dope composition which exhibits thermal phase inversion boundaries at a so-called lower critical solution temperature (LCST) as well as at an upper critical solution temperature (UCST). These properties are exploited by a manufacturing process that employs a temperature-regulated nonsolvent quench bath which serves to initiate the temperature-dependent phase inversion phenomenon as well as freezing or precipitating out and preserving the resultant microporous structure.

Brief Summary Paragraph Right (76):
In conjunction with the procedure disclosed for the production of anisotropic as well as isotropic microporous flat sheet or hollow fiber membranes, the present invention

further describes an improved spinnerette assembly comprised of two independent concentric annuli surrounding a central bore which optionally contains therein a removable hollow pin. This improved spinnerette, which can be maintained at a desired temperature with the aid of means for external heating, is designed to accommodate three separate entry ports for controlling the flow of three separate fluids: namely, a dope composition, an intraannular fluid, and an extraannular fluid. The design of this improved spinnerette is quite simple and economical and has no need for tangential entry ports.

Brief Summary Paragraph Right (77):
The ability to deliver the extraannular fluid over the outer surface of an extruded hollow fiber permits, among other things, the production of hollow fiber membranes with a substantially isotropic microporous structure in all directions throughout the membrane. As disclosed further below, other membrane structures (e.g., skinned, double-skinned, anisotropic) are also possible by the methods of the present invention.

Brief summary Paragraph Center (9):
2.6 Prior Methods For The Production Of Microporous Membranes

Brief Summary Paragraph Type 1 (6):
2.6 Prior Method for the Production of Microporous Membranes

Brief Summary Paragraph Type 1 (19):
6.8 Hollow Fiber Spinning of Relatively Isotropic Microporous Membranes Primarily for Affinity Applications

Brief Summary Paragraph Type 2 (4):
5.3.3 Process for Manufacturing Substantially Isotropic Microporous Membranes

Detailed Description Paragraph Right (2):
Preferably, the apparatus automatically performs the separation process with repeated rapid cycles. The apparatus membrane comprises substantially isotropic microporous membrane material on to which a ligand, specific to the target molecules, is anchored. The apparatus contains pumps and valves controlled by a microprocessor and, in its operation, is programmed to cycle through a membrane loading step with the feed stream containing the target molecule such as a protein, a washing step to remove remaining contaminants, an elution step to recover the target molecule and a membrane regeneration step to complete the cycle. Fluid containing the target molecule is recirculated with a feed pump from a reservoir and through the feed chamber of the apparatus. There, a fraction of the feed perfuses the membrane, regulated by a filtrate pump at a flow rate consistent with the device geometry and membrane characteristics, permitting the target molecule to be captured by the membrane. Eluant is used to flush the filtrate chamber, followed by elution of the target molecule from the membrane bound ligand. The membrane device is then equilibrated with a regeneration buffer by first flushing the filtrate chamber, and then flushing the membrane and feed chamber.

Detailed Description Paragraph Right (11):
This invention utilizes the functionalizable chain ends present in practically all polymeric materials. The instant invention provides that treatment of suitable hydrophobic polymer samples, under heterogeneous conditions, with linker moieties capable of forming a covalent bond with the hydrophobic polymer end groups, allows for the modification of the surface properties of the polymer while preserving desirable bulk properties. Using the methods of the invention, the surface properties of any article manufactured from the subject polymer may be modified while preserving the shape and microstructure of the manufactured article. Thus, bulk polymers with functionalizable end groups may be derivatized or modified under heterogeneous conditions whether the polymer is in powdered form, in the form of an extruded fiber, a microporous membrane, a solid strip, molded into a pipe, or incorporated into an artificial organ, skin, or prosthetic device. Such an article may be manufactured by techniques well-known in the art. Examples of these manufacturing methods include but are not limited to, injection, compression, and blow molding, blowing, calendering, casting, coating, forming, lamination, or extrusion methods.

Detailed Description Paragraph Right (12):
Furthermore, a process for the production of substantially isotropic microporous membranes is disclosed, which process takes advantage of the special properties of a unique four-component dope composition and an improved double annular multi-port spinnerette assembly.

Detailed Description Paragraph Right (19):
The morphology and isotropy of microporous membranes are critical to the performance of hollow fibers employed in affinity purification. Thus, pore sizes in the 0.22 um to several micron range are not expected to result in sieving of protein molecules. Membranes in this pore size range are by convention classified as microfilters (MF) based on their ability to reject particulate matter.

Detailed Description Paragraph Right (22):
As membrane pore size increases (i.e., from UF to MF type membranes), internal surface area inevitably decreases. However, microfilters have the advantage of high volumetric flux at low transmembrane pressure, and a much lower probability of fouling and/or plugging. For these reasons, a MF-type, isotropic microporous hollow fiber is a highly desirable membrane for affinity purification applications.

Detailed Description Paragraph Right (27):
In one preferred embodiment of the invention, flat sheet microporous membranes, comprising polyethersulfone (PES) as the primary or bulk polymer component, are immersed overnight at room temperature in a basic aqueous solution containing a diepoxide linker moiety. Optionally, the membrane samples may be preconditioned by heating them in aqueous solutions or washing them in acetonitrile or isopropanol. The substituted phenol groups of the PES chain ends exposed at the membrane surface are deprotonated by the base giving a nucleophilic phenoxide group. This nucleophile attacks an epoxide group of the linker moiety forming a covalently bound (i.e., ether bond) linker moiety. Because the covalently bound linker moieties are capable of forming at least one other covalent bond (via e.g., a second epoxide group) with another chemical entity, any molecule, macromolecule, or ligand, may then be covalently bound to the membrane surface. The covalently bound macromolecule is thus held very strongly and cannot be removed by washing or other mechanical means.

Detailed Description Paragraph Right (39):
The process for derivatizing hydrophobic polymer interfaces discussed above is especially applicable to the surface modification of microporous membranes. In the course of devising new ways of preparing membranes, the inventors have discovered a unique four-component dope composition which in combination with other aspects of the overall manufacturing process provides membranes with substantially isotropic microporous structures formed either as flat sheets or, perhaps more significantly, hollow fibers.

Detailed Description Paragraph Right (46):
Examples of other suitable polymer pairs which may be utilized in this invention include, but are not limited to: polysulfone (PS)/PEO; PES/Polyvinyl pyrrolidone (PVP) (particularly the high molecular weight forms, e.g., MW about 360,000 of PVP); PS/PVP (MW .about.360,000); Polyvinylidene fluoride (PVDF/PEO; PES/Epichlorohydrin copolymers of PEO; PES/Polyvinyl alcohol (PVA); Polyphenylene oxide (PPO)/Hydrophilized forms of polystyrene (including copolymers and sulfonylated polystyrene); poly(acrylonitrile) (PAN) and copolymers/hydrophilic acrylic polymers (including polyacrylamide), or PVP; PES/hydrophilized forms of PES (including sulfonated PES); and PS/hydrophilized forms of PS.

Detailed Description Paragraph Right (51):
It has also been discovered that membranes with substantially isotropic porous structures (i.e., structures in which the pore diameters are within about an order of magnitude or so of each other) can be prepared and preserved by subjecting the homogeneous dope composition to an abrupt change in temperature, preferably at or above the LCST, and essentially simultaneously "freezing out" the precipitated structure by introducing a nonsolvent for at least the primary polymer component. This procedure is most conveniently carried out in the case of flat sheet membranes, by immersing a liquid film of the dope composition in a nonsolvent quenching bath (e.g., water) maintained at a temperature above the LCST. Quenching the mixture above the LCST produces more open membrane structures with larger isotropic pores in the micron range. By contrast, anisotropic microporous or macrovoid-containing membranes are obtained from quench baths held below the LCST or UCST. The membrane pore sizes, besides being substantially isotropic, may thus be potentially controlled by selecting the temperature of the quench bath. Furthermore, the membranes produced by quenching above the LCST are substantially skinless having a very high density of pores in the exterior surface of the membrane.

Detailed Description Paragraph Right (53):

While current techniques for preparing microporous hollow fibers are capable of producing membranes with surface pores ranging from tenths of a micron to several microns in diameter, such conventional membranes of the prior art typically retain particles more than an order of magnitude smaller than the surface pore size. As an example, the nominal 0.2 .mu.m-rated hollow fiber commercially available from AG Technology (Needham, Mass.) has been found to substantially completely reject latex spheres as small as 0.03 .mu.m; which is more than an order of magnitude smaller than the surface pore size of about 1 .mu.m as revealed by SEM examination. Furthermore, SEM examination reveals that pores rapidly decrease in diameter to less than 0.1 .mu.m below the lumen surface over a distance of a few microns. SEM studies of such a membrane after a 0.03 .mu.m latex challenge test shows entrapment of latex particles within the finely porous region in the matrix below the lumen surface.

Detailed Description Paragraph Right (54):
A typical microporous hollow fiber of the present invention was determined by SEM to contain surface pores in the range of 1 .mu.m. Latex sphere challenge tests, as a means for defining isotropy, show that latex particles as large as 0.25 .mu.m passed freely across the membrane wall. Furthermore, SEM examination of the hollow fiber wall confirmed that pore size distribution across the entire membrane wall was substantially isotropic, with the smallest pores in the fiber wall typically being no smaller than about 0.3 .mu.m.

Detailed Description Paragraph Right (58):
For the production of hollow fiber membranes having substantially isotropic microporous structures, manufacturing procedures more sophisticated than immersing a liquid film of dope composition into a quenching bath (i.e., in the production of flat sheets) are required. For this purpose an improved spinnerette assembly a schematic diagram of which is shown in FIG. 4, is used.

Detailed Description Paragraph Right (68):
Furthermore, although the intra- and extraannular fluids may serve both to initiate the thermal phase separation and to quench the resulting microporous structure, the stationary washing/quenching bath still serves to partially quench and preserve the membrane structure. As mentioned above, the strong solvent is also washed away from the membrane in the wash process along with other contaminants. Preferably, the bath temperature should also be kept above the LCST of the dope composition.

Detailed Description Paragraph Right (69):
Phase boundaries may naturally serve to define process temperatures. Typically, a temperature of about 10.degree. C. above the LCST is employed in producing relatively isotropic microporous membranes with pores in the range of 1 .mu.m in diameter. Dopes can be maintained in the single phase region of the phase diagram (e.g., at 60.degree. C.) before reaching the spinnerette in the extrusion process, or equally useful, the dope may be caused to phase separate either in the dope lines or dope pot. The point up-stream of the spinnerette at which thermal phase inversion occurs does not seem to matter greatly, an observation which is contrary to the general teachings of the membrane art which teaches that dopes should be maintained in the single phase at all times until it emerges from the spinnerette. According to conventional wisdom, phase separation in any part of the spinning apparatus should be avoided because it normally results in irreproducible and inferior membrane properties (e.g., defects, closed cell matrix structure, and the like). It has been found, however, that the important consideration is that the dope attains a temperature equal to or greater than the LCST before or very soon after contacting quench media. Thus in the case of flat sheet casting, polymer dopes are preferably extruded in the single phase and quenched in about 80.degree.-90.degree. C. water. For hollow fibers, both the quench bath and spinnerette are preferably maintained at about 80.degree.-90.degree. C.

Detailed Description Paragraph Right (71):
In extruding preferred PES/PEO dope compositions a number of bore injection (intraannular) fluids can be employed to good effect. These include: water, water/solvent (e.g., NMP) mixtures, pure solvents (e.g., NMP), water soluble polymer solutions (e.g., PVA), gas (e.g., nitrogen), humidified gas, various non-solvents and liquids which are immiscible with components in the dope, according to one's ultimate goal. When making relatively isotropic microporous membranes with surface pores in the range of 1 .mu.m, the preferred bore injection fluid is a water/NMP mixture.

Detailed Description Paragraph Right (135):
Fiber sample 2600-6 is examined by electron microscopy and pores in the 1-3 .mu.m range are observed on the two surfaces. Overall pore size distribution in the matrix of the

approximately 300 .mu.m fiber wall varies within the range of about 1-2 orders of magnitude, but the great majority of the pores are within 1 order of magnitude of each other in size. The results indicate that this membrane is an example of a substantially skinless relatively isotropic microporous membrane. By contrast, fiber 2600-5 (which is made minus the extraannular fluid) is a far more anisotropic microporous membrane structure.

Detailed Description Paragraph Right (137):
A wide range of microfiltration and ultrafiltration applications can be addressed by these membranes (with or without further surface modification or hydrophilization), where the relatively low protein binding surfaces minimize fouling and plugging of the matrix. Of particular interest is the use of the relatively isotropic microporous fibers (e.g., fiber 2600-6) for cell separation. This separation of cells from accompanying liquid can be achieved at very high fluxes without catastrophic decay in hydraulic permeability, which is typically observed for commercially available hollow fibers. Some examples of such cell separation applications include: clarification of cell broth and conditioned media (where affinity binding and clarification may be combined to reduce the number of unit operations in protein purification), and separation of blood cells for medical applications.

Detailed Description Paragraph Left (3):
5.3.3 Process For Manufacturing Substantially Isotropic Microporous Membranes

Detailed Description Paragraph Center (14):
6.8 Hollow Fiber Spinning of Relatively Isotropic Microporous Membranes Primarily For Affinity Applications

Current US Cross Reference Classification (4):
210/500.41

Issued US Cross Reference Classification (4):
210/500.41

Field of Search Class/SubClass (4):
210/500.41

U.S. Reference US Original Classification (22):
210/500.41

CLAIMS:

1. A method for carrying out affinity purification of a ligate in a hollow fiber membrane system comprising:

(a) providing a ligate-containing liquid to a first side of at least one porous hollow fiber membrane with a ligand immobilized thereto, said membrane having a microporous structure, said liquid being under a pressure sufficient to cause a first portion of said liquid to flow convectively and tangentially across said first side of said membrane, and a second portion of said liquid being caused to flow convectively into and through said membrane emerging on a second side of said membrane, wherein said ligate present in said liquid binds to said ligand and is thereby separated from said liquid;

(b) withdrawing said first portion of said liquid from said first side;

(c) recirculating said first portion of said liquid to said first side of said membrane;

(d) repeating steps (a) to (c) until a majority of said liquid has flowed into and through said membrane; and

(e) providing an elution solution to one side of said membrane under a pressure sufficient to cause said elution solution to flow into and through said membrane and to effect the disassociation of any ligate-ligand bonds formed in step (a) wherein any ligate bound to said ligand is eluted with said elution solution.

25. A method for carrying out affinity purification of a ligate in a hollow fiber membrane system comprising:

(a) providing a ligate-containing liquid to a first side of at least one microporous hollow fiber defining a membrane, said membrane having a ligand immobilized thereto, said liquid being under a pressure sufficient to cause a first portion of said liquid to flow convectively and tangentially across said first side of said membrane, with a second portion of said liquid being caused to flow convectively into and through said membrane emerging on a second side of said membrane, wherein said ligate present in said liquid binds to said ligand and is thereby separated from said liquid;

(b) withdrawing said first portion of said liquid from said first side;

(c) recirculating said withdrawn first portion of said liquid to said first side of said membrane;

(d) repeating steps (a) to (c) until a majority of said liquid has flowed to the second side; and

(e) providing an elution solution to one side of said membrane under a pressure sufficient to cause said elution solution to flow into and through said membrane and to effect the disassociation of any ligate-ligand bonds, wherein any ligate bound to said ligand is eluted with said elution solution.

26. An apparatus for carrying out affinity separation comprising:

at least one porous hollow fiber membrane having a ligand immobilized thereto;

means for enclosing said at least one porous hollow fiber membrane;

means for providing a fluid in intimate contact with a first side of said enclosed porous hollow fiber membrane;

first exit means for directing into a first container any fluid present on a second side of said enclosed porous hollow fiber membrane opposite the first side to which said fluid is first provided according to the fluid providing means; and

second exit means for directing said fluid present on the first side of said membrane into a second container,

wherein at least part of said fluid leaving from said first exit means has originated from said fluid providing means and said hollow fiber membrane has a substantially isotropic microporous structure in all directions throughout the membrane, with pores large enough to permit convective flow of macromolecule-containing solutions across the hollow fiber membrane.

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L3	L2 and water same flow	6	L3
L2	L1 and microfiltration	11	L2
L1	polysulfone same polyvinylalcohol and hollow fiber	31	L1

END OF SEARCH HISTORY

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L21: Entry 1 of 2 File: USPT Apr 4, 2000

DOCUMENT-IDENTIFIER: US 6045694 A
TITLE: Cationically charge-modified membranes

Brief Summary Paragraph Right (5):
Ultrafiltration and microfiltration membranes utilized in industry, particularly in the food processing industry and in environmental applications, are typically hydrophobic membranes which may be surface-modified with a hydrophilic material to reduce fouling and to confer additional desirable properties to the membrane. Membranes may be isotropic or asymmetric (anisotropic) in their pore structure. Isotropic membranes have a uniform pore structure throughout the membrane. Asymmetric membranes do not have a uniform pore structure throughout the membrane. Asymmetric porous membranes are distinguished from isotropic, homogeneous membrane structures whose flow and retention properties are independent of flow direction. Asymmetric membranes are useful in microfiltration, ultrafiltration, and reverse osmosis processes.

Brief Summary Paragraph Right (20):
The hydrophobic polymer of the membrane may advantageously be a sulfone polymer, polyvinylidene difluoride, polytetrafluoroethylene, polypropylene, or polyethylene. Preferred sulfone polymers are polysulfone, polyarylsulfone, and polyethersulfone.

Brief Summary Paragraph Right (25):
The hydrophobic polymer of the membrane may advantageously be a sulfone polymer, polyvinylidene difluoride, polytetrafluoroethylene, polypropylene, or polyethylene. Preferred sulfone polymers are polysulfone, polyarylsulfone, and polyethersulfone.

Brief Summary Paragraph Right (28):
A third aspect of the invention provides a positively charged polymer membrane. This membrane is cast from a formulation that includes a sulfone polymer and a copolymer of vinylpyrrolidone and a cationic imidazolinium compound. The formulation also includes a low molecular weight organic acid and a solvent. The sulfone polymer may be polysulfone, polyarylsulfone or polyethersulfone. The cationic imidazolinium compound may advantageously be methylvinylimidazoliummethyl sulfate. The acid may be selected from the group consisting of formic, acetic, propionic and butyric acid, and preferred solvents are N-methylpyrrolidone or dimethylformamide.

Brief Summary Paragraph Right (31):
The sulfone polymer may be selected from the group consisting of polysulfone, polyarylsulfone and polyethersulfone. A preferred cationic imidazolinium compound is methylvinylimidazoliummethyl sulfate. The acid may be selected from the group consisting of formic, acetic, propionic and butyric acid, and the solvent may advantageously be N-methylpyrrolidone or dimethylformamide.

Detailed Description Paragraph Right (4):
It has been discovered that in the post-treatment of formed membranes or in the quench or rinse bath treatment of cast membranes, it is not necessary to utilize a latently reactive polymeric wetting agent such as polyvinylpyrrolidone or polyethylene glycol or an activated modifying polymer such as a chlorohydrin derivative of hydroxypropylcellulose to render the initially hydrophobic membrane irreversibly hydrophilic. It has been discovered that an initially hydrophobic membrane may be made irreversibly hydrophilic through a simple contacting process with the improved polymeric wetting agents of the present invention, or alternatively with a contacting process followed by a simple heating process. Polymeric wetting agents are selected from the group consisting of polyvinylpyrrolidone and copolymers of pyrrolidone, hydroxypropylcellulose (HPC), hydroxypropylmethylcellulose, methylcellulose (Methocell.TM.) and other cellulose polymers with hydrophilic functional groups, and

polyvinyl alcohol (PVA), with HPC being particularly preferred.

Detailed Description Paragraph Right (5):
In the method of the present invention for cationic charge modification of a formed initially hydrophobic membrane, the membrane is made hydrophilic with any of the improved polymeric wetting agents of the present invention, following which the membrane is simply contacted simultaneously with a first and second charge-modifying agent in aqueous solution for a brief period, following which the membrane is dried under thermal conditions designed to induce crosslinking which reduces leaching of the first and second charge-modifying agents from the membrane. The first cationic charge-modifying agent may be a polyamine, such as hydroxyethylated polyethyleneimine (HEPEI) or an aziridine-ethylene oxide copolymer. The second cationic charge-modifying agent may be either a high or low molecular weight epichlorohydrin-modified highly branched polyamine. Such polyamines preferably include the high molecular weight KYMENE 736 and KYMENE 450 resins and the RETEN 201 (50,000 daltons) low molecular weight resin. Such polyamines rely upon chemical crosslinking through the epichlorohydrin functional groups to achieve irreversible cationic charge modification, unlike the first cationic charge-modifying agents of the present invention. The molecular weight of the polyamine is typically selected based on the "openness" of the formed membrane. For example, higher molecular weight polyamines are preferably utilized in connection with relatively large pored microfiltration sheet or hollow fiber membranes or melt blown materials, while lower molecular weight compounds are utilized in connection with "tighter" pored membranes, such as membranes having pore sizes less than 0.02 .mu.m. In addition, it has been discovered that if the polyamine is subjected to mild shear conditions prior to contact with the membrane, this assists in lowering the effective molecular weight of the polyamine polymer, perhaps by unwinding, disentangling, or breaking the polymer chains of the higher molecular weight polyamines.

Detailed Description Paragraph Right (9):
Formed membranes that are suitable for wetting and charge modification in accordance with the present invention include virtually any formed initially hydrophobic polymer membrane that has sufficient porosity so as to allow treatment with the wetting and cationic charge-modifying agents or agent. Formed membranes are initially hydrophobic and are rendered hydrophilic through surface treatment with an effective amount of a polymeric wetting agent. More than one polymeric wetting agent may also be employed simultaneously. A wetting agent as used herein has the ability to cause a surface to have increased wettability by water. The wetting of solid surfaces by liquids is an important process for many industrial applications. Wetting, by definition, is the process of one fluid displacing another fluid at a solid surface. However, in most cases, the term is used to describe the displacement of air by a liquid. Generally, suitable wetting agents will contain hydrophilic chemical functional groups such as hydroxyl groups, carboxylic acid groups and the like. Polymeric wetting agents are selected from the group consisting of HPC, hydroxypropylmethylcellulose, Methocell.TM. and other cellulose polymers with hydrophilic functional groups, and PVA, with HPC being particularly preferred.

Detailed Description Paragraph Right (12):
Preferred formed hydrophobic membranes include sheet and hollow fiber cast polymer membranes and melt blown polymer membranes. Membranes in accordance with this aspect of the invention typically possess porosities characteristic of ultrafiltration or microfiltration membranes, with pore sizes ranging from about 0.00021 .mu.m to about 10 .mu.m, and preferably from about 0.01 .mu.m to about 10 .mu.m. Viewed from another perspective, suitable membranes within the ultrafiltration range generally possess molecular weight cutoffs of from about 10,000 daltons to about 100,000 daltons and have pore sizes from 0.00021 to 0.0048 .mu.m, whereas microfiltration membranes typically possess pore sizes of from at least about 0.01 .mu.m through about 10 .mu.m.

Detailed Description Paragraph Right (13):
Any hydrophobic polymer that can be formed into a membrane by a casting, melt-blowing or other process, that possesses the pore size criteria described above and which can be rendered hydrophilic through treatment with the wetting agents described above, is generally acceptable in accordance with this aspect of the invention. Preferred polymers include sulfone polymers, such as polysulfone, polyarylsulfone, and polyethersulfone, fluorinated polymers, such as polyvinylidene difluoride (PVDF) and polytetrafluoroethylene (PTFE), polypropylene, and others such as polyethylene. Cast membranes are generally formed from sulfone polymers whereas melt blown membranes are generally formed from polypropylene or polyethylene. Cast membranes are preferably formed, without limitation, in accordance with Wrasidlo U.S. Pat. No. 4,629,563, or co-pending U.S. patent application Ser. Nos. 08/473,206, filed on Jun. 7, 1995;

08/476,189, filed Jun. 7, 1995; 08/484,216, filed on Jun. 7, 1995; 08/498,722, filed on Jul. 5, 1995; 08/661,839, filed Jun. 11, 1996; and 08/785,962, filed on Jan. 22, 1997. Melt blown membranes are preferably formed in accordance with U.S. patent application Ser. No. 08/433,006, filed on May 2, 1995. As such, preferred membranes utilized in accordance with the invention may possess pore size gradients through the cross-section of the membrane.

Detailed Description Paragraph Right (21):
With cast membranes, as will be appreciated, treatment with HPC can be conveniently accomplished in a quench or rinse bath immediately or shortly after casting. By way of example, a polysulfone based Wrasidlo-type unstable dispersion phase inversion formulation (see U.S. Pat. No. 4,629,563, Example II), can be cast onto an inert support and quenched using an aqueous bath including HPC (approximately 0.01 w/v % to about 0.5 w/v %) to form a highly anisotropic microfiltration membrane. The resulting membrane, following drying, is inherently hydrophilic, owing to the HPC treatment. A similar process can be utilized for the preparation of hollow fiber membranes.

Detailed Description Paragraph Right (28):
Further aspects of the invention provide a cationic charge-modified membrane and a process to prepare such a membrane by casting in a film a mixed polymer solution containing a sulfone polymer, a copolymer of vinylpyrrolidone and a cationic imidazolinium compound, a low molecular weight organic acid and a solvent, quenching the resulting film in an aqueous bath, and washing and drying the coagulated membrane. Conventional film casting, quenching, rinsing and drying procedures are employed. In a preferred embodiment, the sulfone polymer may be selected from the group consisting of polysulfone, polyarylsulfone and polyethersulfone. Polyethersulfone is preferred. Polyethersulfone can be employed with the chemical structure and molecular weight range as described in U.S. Pat. No. 5,531,893 to Hu, et al., such disclosure being incorporated herein by reference. Generally, a concentration between about 5 and about 50% by weight of sulfone polymer may be employed in the polymer solution. Preferably, a concentration between about 10 and about 25% by weight is employed. Most preferably, a concentration of about 15% by weight of sulfone polymer is employed. The copolymer of vinylpyrrolidone and a cationic imidazolinium compound may be any copolymer containing any number of repeating vinylpyrrolidone groups and imidazolinium groups. In a preferred embodiment, the copolymer of vinylpyrrolidone and a cationic imidazolinium compound is a terpolymer of vinyl caprolactam, vinylpyrrolidone and methylvinylimidazoliummethyl sulfate. Generally, a concentration between about 0.5 and about 10% by weight of copolymer may be employed in the polymer solution. Preferably, a concentration between about 1.0 and about 5% by weight is employed. Most preferably, a concentration of 1.0-2.0% by weight of copolymer is employed. In another preferred embodiment, the low molecular weight organic acid is selected from the group consisting of formic, acetic, propionic and butyric acid. Propionic acid is preferred. Generally, a concentration between about 10 and about 60% by weight of acid may be employed in the polymer solution. Preferably, a concentration between about 25 and about 45% by weight is employed. Most preferably, a concentration of about 34-35% by weight of acid is employed. In another preferred embodiment, the solvent is N-methylpyrrolidone or dimethylformamide. N-methylpyrrolidone is preferred. Generally, a concentration between about 10 and about 60% by weight of solvent may be employed in the polymer solution. Preferably, a concentration between about 25 and about 55% by weight is employed. Most preferably, a concentration of about 49% by weight of solvent is employed. In another embodiment, the mixed polymer solution may be 5-50 w/w % sulfone polymer and 0.5-10.0 w/w % copolymer of vinylpyrrolidone and a vinylimidazole compound. In an additional embodiment, the mixed polymer solution may be 10-25 w/w % polyethersulfone and 1.0-5.0 w/w % copolymer of vinylpyrrolidone and methylvinylimidazoliummethyl sulfate.

Detailed Description Paragraph Right (36):
Several polysulfone membrane samples (0.2 .mu.m average surface pore size, available from Memtec America Corporation), obtained from roll stock that had previously been rendered hydrophilic by treatment with HPC, were charge-modified in accordance with the invention. HPC treatment was accomplished as previously described in a quench bath during the manufacture of the cast membranes. The concentration of the HPC in the quench bath was approximately 0.04%.

Detailed Description Paragraph Right (40):
A 5 .mu.m polysulfone membrane that was rendered hydrophilic through treatment with HPC as described above was then charge-modified in accordance with the invention, as follows: The membrane was dipped into a charge-modifying solution containing 0.8% HEPEI and 1.2% KYMENE 736 having a pH of between about 8 and about 8.5 for about 30 seconds. The still wet membrane was passed through a 115.degree. C. oven for three minutes to

effect crosslinking.

Detailed Description Paragraph Right (42):
Three microporous polysulfone membrane samples (0.1 .mu.m average surface pore size, available from Memtec America Corporation), rendered hydrophilic with HPC in accordance with the invention, were utilized in a comparative test. One membrane sample was kept untreated (5a), a second membrane was charge-modified via dip-coating in a solution containing 1% HEPEI and 1.2% KYMENE 736 (Sb), and a third membrane sample was charge-modified via dip-coating in a solution containing 0.5% HEPEI and 0.6% KYMENE 736 at a pH of about 8 to about 8.5 (5c). Following treatment, while still wet, Samples 5b and 5c were passed through an oven at about 115.degree. C. for about four minutes to effect crosslinking.

Detailed Description Paragraph Right (45):
Four microporous polysulfone membrane samples (0.2 .mu.m average surface pore size, BTS-80, available from Memtec America Corporation), rendered hydrophilic with HPC in accordance with the invention, were utilized in a comparative test. One membrane sample was kept untreated (6a), and a second membrane was charge-modified by dip-coating in a solution containing 1% HEPEI and 1.2% KYMENE 736 (6b). A third membrane sample was charge-modified by dip-coating in a solution containing 0.1% HEPEI and 0.12% KYMENE 736 (6c), and a fourth membrane sample was treated with 0.2% poly G-20 and 0.2% KYMENE 736 at a pH of about 8 to about 8.5 (6d). Following treatment, while still wet, Samples 6b, 6c and 6d were passed through an oven at about 115.degree. C. for about four minutes to effect crosslinking.

Detailed Description Paragraph Left (10):
Charge Modification of Very Large Pored Microfiltration Membranes

Detailed Description Paragraph Left (11):
Comparison of HPC-Treated Polysulfone Membranes Versus HPC-Treated and Charge-modified Polysulfone Membranes

Detailed Description Paragraph Left (12):
Comparison of HPC-Treated Polysulfone Membranes Versus HPC-Treated and Charge-modified Polysulfone Membranes

Current US Cross Reference Classification (5):
210/500.41

Issued US Cross Reference Classification (4):
210/500.41

Field of Search Class/SubClass (5):
210/500.41

CLAIMS:

15. The membrane of claim 14, wherein said sulfone polymer is selected from the group consisting of polysulfone, polyarylsulfone and polyethersulfone.

28. The method of claim 27, wherein said sulfone polymer is selected from the group consisting of polysulfone, polyarylsulfone and polyethersulfone.

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☐ 11. Document ID: US 4877529 A

L43: Entry 11 of 16 File: USPT Oct 31, 1989

US-PAT-NO: 4877529
DOCUMENT-IDENTIFIER: US 4877529 A

TITLE: Separation of organic liquids

DATE-ISSUED: October 31, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Pasternak; Mordechai	Spring Valley	NY		
Bartels; Craig R.	Wappinger Falls	NY		
Reale, Jr.; John	Wappinger Falls	NY		

US-CL-CURRENT: 210/500.37; 210/651, 210/654

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

☐ 12. Document ID: US 4755299 A

L43: Entry 12 of 16 File: USPT Jul 5, 1988

US-PAT-NO: 4755299
DOCUMENT-IDENTIFIER: US 4755299 A

TITLE: Multi-layer membrane and the use thereof for the separation of liquid mixtures according to the pervaporation process

DATE-ISSUED: July 5, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Bruschke; Hartmut	Nussloch			DEX

US-CL-CURRENT: 210/640; 210/321.84, 210/500.32, 210/500.41, 210/500.42, 210/500.43, 95/45

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

☐ 13. Document ID: US 4559139 A

L43: Entry 13 of 16 File: USPT Dec 17, 1985

US-PAT-NO: 4559139
DOCUMENT-IDENTIFIER: US 4559139 A

TITLE: High performance semipermeable composite membrane and process for producing the same

DATE-ISSUED: December 17, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Uemura; Tadahiro	Kyoto			JPX
Kurihara; Masaru	Otsu			JPX

US-CL-CURRENT: 210/490; 210/500.41, 427/245

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

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☐ 14. Document ID: US 4387024 A

L43: Entry 14 of 16 File: USPT Jun 7, 1983

US-PAT-NO: 4387024
DOCUMENT-IDENTIFIER: US 4387024 A

TITLE: High performance semipermeable composite membrane and process for producing the same

DATE-ISSUED: June 7, 1983

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kurihara; Masaru	Ohtsu			JPX
Uemura; Tadahiro	Kyoto			JPX
Okada; Kiyoshi	Kusatsu			JPX

US-CL-CURRENT: 210/490; 210/500.28, 210/500.41, 427/245

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 15. Document ID: US 4250029 A

L43: Entry 15 of 16 File: USPT Feb 10, 1981

US-PAT-NO: 4250029
DOCUMENT-IDENTIFIER: US 4250029 A

TITLE: Coated membranes

DATE-ISSUED: February 10, 1981

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kiser; Ernest J.	Iowa City	IA		
Latty; James A.	Seal Beach	CA		

US-CL-CURRENT: 210/652; 210/490, 210/500.34, 210/500.35, 210/500.41, 210/650, 210/654

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 16. Document ID: US 3940469 A

L43: Entry 16 of 16 File: USPT Feb 24, 1976

US-PAT-NO: 3940469
DOCUMENT-IDENTIFIER: US 3940469 A

TITLE: Process for forming hollow fibers

DATE-ISSUED: February 24, 1976

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Steigelmann; Edward F.	Park Forest	IL		
Hughes; Robert D.	Park Forest	IL		
Gabor; Joseph	Whiting	IN		

US-CL-CURRENT: 264/209.1; 210/500.23, 210/500.38, 210/500.41, 264/205, 428/398, 521/61, 525/58, 96/10

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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JPO Abstracts Database
EPO Abstracts Database
Derwent World Patents Index
IBM Technical Disclosure Bulletins

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L29	5024765.pn.	1	L29
L28	5462667.pn.	1	L28
L27	5462867.pn.	1	L27
L26	5464538.pn.	1	L26
L25	5503746.pn.	1	L25
L24	5531893.pn.	1	L24
L23	5543054.pn.	1	L23
L22	5633300.pn.	1	L22
L21	l5 and polysulfone and pva and microfiltration	2	L21
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L19	L18 and pores same size	0	L19
L18	L17 and hydrophilic	6	L18
L17	polysulfone and microporous	77	L17
L16	L15 and microporous	0	L16
L15	L5 and microfiltration	0	L15
L14	l5 and microfiltration and pores and hydrophilic	0	L14
L13	polysulfone and microprorous and hydrophilic	0	L13
L12	membrane and large pore and microfiltration	0	L12
L11	"stengaard"	1	L11
L10	"5091086"	0	L10
L9	polysulfone same pva	20	L9
DB=USPT; PLUR=YES; OP=ADJ			
L8	l5 and pva same polysulfone	6	L8
L7	L6 and pva same polysulfone	3	L7
L6	L5 and pva and microporous	9	L6
L5	210/500.41	455	L5
L4	L2 and pore size	11	L4
L3	L2 and water same flow	6	L3
L2	L1 and microfiltration	11	L2
L1	polysulfone same polyvinylalcohol and hollow fiber	31	L1

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1. Document ID: US 6440309 B1

L3: Entry 1 of 23 File: USPT Aug 27, 2002

US-PAT-NO: 6440309
DOCUMENT-IDENTIFIER: US 6440309 B1

TITLE: Ceramic-supported polymer (CSP) pervaporation membrane

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cohen; Yoram	Los Angeles	CA	90064	

US-CL-CURRENT: 210/640; 210/490, 210/500.27, 210/500.42, 95/45, 96/4

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

2. Document ID: US 6306594 B1

L3: Entry 2 of 23 File: USPT Oct 23, 2001

US-PAT-NO: 6306594
DOCUMENT-IDENTIFIER: US 6306594 B1

TITLE: Methods for microdispensing patterened layers

DATE-ISSUED: October 23, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Lauks; Imants R.	Yardley	PA		
Mier; Randall M.	Morrisvile	PA		
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Van der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		

US-CL-CURRENT: 435/6; 430/127, 430/14, 430/16, 430/4, 430/5, 430/56, 430/96, 430/97, 435/174, 435/180, 435/4, 435/5, 436/518, 436/524, 436/525, 436/531

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L3: Entry 4 of 23 File: USPT Nov 30, 1999

DOCUMENT-IDENTIFIER: US 5993661 A
**** See image for Certificate of Correction ****
TITLE: Macroporous or microporous filtration membrane, method of preparation and use

Abstract Text (1):
This invention relates to a microporous or macroporous affinity filtration membrane wherein the matrix is composed of chitosan or chitin and the pores are made by dissolution of porogen during the preparation of the membrane. The invention also relates to a method of preparation of the membrane comprising preparing an acidic chitosan solution containing porogen, shaping the suspension into a membrane, and dissolving the porogen by immersing the membrane in an alkaline solution. To prepare chitin membranes, the chitosan membranes are acetylated. The special feature of the membrane is that the pore size can be controlled by varying the size of the porogen. The membranes are suitable for affinity purification of macromolecules.

Brief Summary Text (3):
The present invention relates to the area of affinity purification of macromolecules. More particularly, the invention provides an affinity membrane, wherein the pore size is based upon the size of the porogen selected, a method for preparation of the membrane, and a method for affinity purification of macromolecules.

Brief Summary Text (5):
Affinity membrane filtration (AMF) has recently emerged as an alternative to affinity column chromatography. An advantage of AMF is that high flow rates at low pressure drops can be achieved, thereby greatly improving the washing, elution, and regeneration processes, and decreasing the probability of deactivation of the biomolecules by shortening their exposure to an unfavorable medium.

Brief Summary Text (6):
The key to efficient AMF is the preparation of the affinity membranes. In general, two approaches have been employed to prepare affinity membranes. In the most common method, microporous affinity membranes are prepared from polyethylene, polypropylene, nylon, polysulfone, and glass. However, these membranes are usually hydrophobic and relatively inert, and hence require modifications. In addition, some of the membranes may require amplification of the number of active groups. To overcome these drawbacks, a second approach has been employed wherein membranes are prepared that have preincorporated functional groups. However, the problems with this type of membranes include hydrophobicity (poly glycidyl methacrylate-co-ethylene dimethacrylate membrane), brittleness, and solubility in acids (cellulose acetate membrane). Another drawback with both of the above methods is that the pore size of the membrane cannot be easily controlled.

Brief Summary Text (7):
Recently chitosan membranes have been suggested as affinity membranes for immobilization of various macromolecules having affinity for chitosan. Next to cellulose, chitin (poly (N-acetyl-D-glucosamine)), is the most abundant biopolymer. Chitosan, the deacetylated form of chitin, is soluble in dilute aqueous organic acids but is insoluble in alkaline solutions. Chitosan molecules contain a large number of reactive hydroxyl and amine groups, which can easily attach ligands. In view of its hydrophilicity, excellent film-forming ability, good mechanical properties, and high chemical reactivity (containing hydroxyl and amine groups),

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 3. Document ID: US 6174443 B1

L3: Entry 3 of 23 File: USPT Jan 16, 2001

US-PAT-NO: 6174443
DOCUMENT-IDENTIFIER: US 6174443 B1
**** See image for Certificate of Correction ****

TITLE: Purification of wheat germ agglutinin using macroporous or microporous filtration membrane

DATE-ISSUED: January 16, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ruckenstein; Eli	Amherst	NY		
Zeng; Xianfang	Cary	NC		

US-CL-CURRENT: 210/651; 127/34, 210/638, 210/650, 530/414

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 4. Document ID: US 5993661 A

L3: Entry 4 of 23 File: USPT Nov 30, 1999

US-PAT-NO: 5993661
DOCUMENT-IDENTIFIER: US 5993661 A
**** See image for Certificate of Correction ****

TITLE: Macroporous or microporous filtration membrane, method of preparation and use

DATE-ISSUED: November 30, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ruckenstein; Eli	Amherst	NY		
Zang; Xianfang	Buffalo	NY		

US-CL-CURRENT: 210/651; 210/231, 210/490, 210/500.23, 210/500.29, 210/500.42, 210/636, 264/200, 264/41

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 5. Document ID: US 5837454 A

L3: Entry 5 of 23 File: USPT Nov 17, 1998

US-PAT-NO: 5837454
DOCUMENT-IDENTIFIER: US 5837454 A

TITLE: Process for the manufacture of wholly microfabricated biosensors

DATE-ISSUED: November 17, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Lauks; Imants R.	Yardley	PA		
Mier, deceased; Randall M.	late of Morrisvile	PA		
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		
Steiner; Susan	Trenton	NJ		
Itak; Jeanne	West Windsor	NJ		

US-CL-CURRENT: 435/6; 204/403.06, 204/411, 204/412, 204/414, 204/415, 204/416, 204/417, 204/419, 422/57, 422/82.08, 427/2.11, 427/2.13, 427/58, 427/96, 435/14, 435/177, 435/25, 435/287.1, 435/287.2, 435/287.9, 435/817, 436/518, 436/524, 436/528, 436/806

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 6. Document ID: US 5837446 A

L3: Entry 6 of 23 File: USPT Nov 17, 1998

US-PAT-NO: 5837446
DOCUMENT-IDENTIFIER: US 5837446 A

TITLE: Process for the manufacture of wholly microfabricated biosensors

DATE-ISSUED: November 17, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Itak; Jeanne	West Windsor	NJ		
Lauks; Imants R.	Yardley			CA
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Steiner; Susan	Trenton	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		
Mier, deceased; Randall M.	late of Morrisville	PA		

US-CL-CURRENT: 435/6; 204/400, 204/411, 204/412, 204/413, 422/57, 422/82.08, 435/14,

435/177, 435/25, 435/287.1, 435/287.2, 435/817, 436/149, 436/150, 436/151, 436/518, 436/528, 436/531, 436/532, 436/806

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 7. Document ID: US 5554339 A

L3: Entry 7 of 23 File: USPT Sep 10, 1996

US-PAT-NO: 5554339
DOCUMENT-IDENTIFIER: US 5554339 A
**** See image for Certificate of Correction ****

TITLE: Process for the manufacture of wholly microfabricated biosensors

DATE-ISSUED: September 10, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Lauks; Imants R.	Yardley	PA		
Mier; Randall M.	Morrisvile	PA		
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		

US-CL-CURRENT: 422/50; 422/63, 422/68.1, 422/69, 422/78, 422/79, 422/82.05, 435/6, 436/501

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 8. Document ID: US 5496637 A

L3: Entry 8 of 23 File: USPT Mar 5, 1996

US-PAT-NO: 5496637
DOCUMENT-IDENTIFIER: US 5496637 A

TITLE: High efficiency removal of low density lipoprotein-cholesterol from whole blood

DATE-ISSUED: March 5, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parham; Marc E.	Bedford	MA		
Duffy; Richard L.	Cambridge	MA		
Nicholson; Donald T.	Leominster	MA		

US-CL-CURRENT: 428/376; 210/500.23, 210/500.35, 210/500.41, 428/398

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 9. Document ID: US 5466575 A

L3: Entry 9 of 23 File: USPT Nov 14, 1995

US-PAT-NO: 5466575
DOCUMENT-IDENTIFIER: US 5466575 A

TITLE: Process for the manufacture of wholly microfabricated biosensors

DATE-ISSUED: November 14, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Lauks; Imants R.	Yardley	PA		
Mier; Randall M.	Morrisvile	PA		
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		

US-CL-CURRENT: 435/6; 204/403.1, 204/403.11, 204/403.12, 204/411, 204/412, 204/414, 204/415, 204/416, 204/417, 204/418, 204/419, 204/430, 204/431, 204/432, 422/82.01, 427/2.13, 427/96, 430/315, 435/177, 435/817, 436/149, 436/806, 438/1, 438/107, 438/110, 438/49, 438/67

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 10. Document ID: US 5420047 A

L3: Entry 10 of 23 File: USPT May 30, 1995

US-PAT-NO: 5420047
DOCUMENT-IDENTIFIER: US 5420047 A
**** See image for Certificate of Correction ****

TITLE: Method for carrying out immunodiagnostic tests

DATE-ISSUED: May 30, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Brandt; Heinz-Dieter	Krefeld			DE
Dhein; Rolf	Krefeld			DE
Hildenbrand; Karlheinz	Krefeld			DE
Stocker; Ronald	Hilden			DE

US-CL-CURRENT: 435/7.9; 422/56, 435/28, 435/4, 435/7.92, 435/805, 435/970, 436/518, 436/531, 436/810

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☐ 11. Document ID: US 5418061 A

L3: Entry 11 of 23 File: USPT May 23, 1995

US-PAT-NO: 5418061
DOCUMENT-IDENTIFIER: US 5418061 A

TITLE: Microporous polysulfone supports suitable for removal of low density lipoprotein-cholesterol

DATE-ISSUED: May 23, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parham; Marc E.	Bedford	MA		
Duffy; Richard L.	Cambridge	MA		

US-CL-CURRENT: 428/398; 428/364

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

☐ 12. Document ID: US 5304307 A

L3: Entry 12 of 23 File: USPT Apr 19, 1994

US-PAT-NO: 5304307
DOCUMENT-IDENTIFIER: US 5304307 A

TITLE: Chargedasymmetric mosaic membrances

DATE-ISSUED: April 19, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/490; 210/500.27, 210/500.29, 210/500.34, 210/500.37, 210/500.38, 210/500.39, 210/500.4, 210/500.41, 210/500.43, 210/651

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

☐ 13. Document ID: US 5265734 A

L3: Entry 13 of 23 File: USPT Nov 30, 1993

US-PAT-NO: 5265734
DOCUMENT-IDENTIFIER: US 5265734 A

TITLE: Silicon-derived solvent stable membranes

DATE-ISSUED: November 30, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monosson			IL
Perry; Mordechai	Petach Tikva			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/654; 210/500.27

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 14. Document ID: US 5258149 A

L3: Entry 14 of 23 File: USPT Nov 2, 1993

US-PAT-NO: 5258149
DOCUMENT-IDENTIFIER: US 5258149 A

TITLE: Process of making a membrane for high efficiency removal of low density lipoprotein-cholesterol from whole blood

DATE-ISSUED: November 2, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parham; Marc E.	Bedford	MA		
Duffy; Richard L.	Cambridge	MA		
Nicholson; Donald T.	Leominster	MA		

US-CL-CURRENT: 264/41; 264/102, 264/184, 264/209.1, 264/211, 264/211.17, 264/233, 264/235

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 15. Document ID: US 5236644 A

L3: Entry 15 of 23 File: USPT Aug 17, 1993

US-PAT-NO: 5236644
DOCUMENT-IDENTIFIER: US 5236644 A

TITLE: Process of making membrane for removal of low density lipoprotein-cholesterol from whole blood

DATE-ISSUED: August 17, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parham; Marc E.	Bedford	MA		
Duffy; Richard L.	Cambridge	MA		
Nicholson; Donald T.	Leominster	MA		

US-CL-CURRENT: 264/41; 264/102, 264/184, 264/209.1, 264/211, 264/211.17, 264/233, 264/235

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 16. Document ID: US 5212050 A

L3: Entry 16 of 23 File: USPT May 18, 1993

US-PAT-NO: 5212050
DOCUMENT-IDENTIFIER: US 5212050 A

TITLE: Method of forming a permselective layer

DATE-ISSUED: May 18, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mier; Randall M.	Ottawa, Ontario			CA
Piznik; Sylvia	Jackson	NJ	08527	
Lauks; Imants R.	Yardley	PA	19067	
Davis; Graham	Plainsboro	NJ	08536	

US-CL-CURRENT: 430/320; 422/930, 430/311, 430/313, 430/325, 430/326, 430/328, 430/330, 435/287.9

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 17. Document ID: US 5205934 A

L3: Entry 17 of 23 File: USPT Apr 27, 1993

US-PAT-NO: 5205934
DOCUMENT-IDENTIFIER: US 5205934 A

TITLE: Silicone-derived solvent stable membranes

DATE-ISSUED: April 27, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monosson			IL
Perry; Mordechai	Petach Tikva			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/500.43; 264/45.1

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 18. Document ID: US 5200051 A

L3: Entry 18 of 23 File: USPT Apr 6, 1993

US-PAT-NO: 5200051
DOCUMENT-IDENTIFIER: US 5200051 A

TITLE: Wholly microfabricated biosensors and process for the manufacture and use thereof

DATE-ISSUED: April 6, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Nepean			CA
Davis; Graham	Plainsboro	NJ		
Itak; Jeanne A.	North Brunswick	NJ		
Lauks; Imants R.	Yardley	PA		
Mier; Randall M.	Morrisville	PA		
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Steiner; Susan J.	Trenton	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Plainsboro	NJ		

US-CL-CURRENT: 204/403.07; 204/403.09, 204/403.1, 204/403.11, 204/403.13, 204/415, 205/778, 205/782.5, 257/253, 422/930 , 435/287.9

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

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☐ 19. Document ID: US 5187010 A

L3: Entry 19 of 23 File: USPT Feb 16, 1993

US-PAT-NO: 5187010
DOCUMENT-IDENTIFIER: US 5187010 A

TITLE: Membrane having high affinity for low density lipoprotein-cholesterol from whole blood

DATE-ISSUED: February 16, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parham; Marc E.	Bedford	MA		
Duffy; Richard L.	Cambridge	MA		

US-CL-CURRENT: 428/398; 210/500.23, 428/364, 428/373, 428/378, 428/400, 521/27

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 20. Document ID: US 5063081 A

L3: Entry 20 of 23 File: USPT Nov 5, 1991

US-PAT-NO: 5063081
DOCUMENT-IDENTIFIER: US 5063081 A
**** See image for Certificate of Correction ****

TITLE: Method of manufacturing a plurality of uniform microfabricated sensing devices having an immobilized ligand receptor

DATE-ISSUED: November 5, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cozzette; Stephen N.	Hightstown	NJ		
Davis; Graham	Plainsboro	NJ		
Itak; Jeanne	Hamilton	NJ		
Lauks; Imants R.	Yardley	PA		
Mier; Randall M.	Ottawa			CA
Piznik; Sylvia	Jackson	NJ		
Smit; Nicolaas	Hightstown	NJ		
Steiner; Susan	Trenton	NJ		
Van Der Werf; Paul	Princeton Junction	NJ		
Wieck; Henry J.	Brooklyn	NY		

US-CL-CURRENT: 435/4; 204/403.1, 204/403.11, 204/415, 204/418, 257/253, 422/57, 422/930, 427/2.13, 427/407.1, 427/414, 435/7.1, 600/345

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC
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☐ 21. Document ID: US 5049282 A

L3: Entry 21 of 23 File: USPT Sep 17, 1991

US-PAT-NO: 5049282
DOCUMENT-IDENTIFIER: US 5049282 A

TITLE: Semipermeable composite membranes

DATE-ISSUED: September 17, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/654

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

☐ 22. Document ID: US 5039421 A

L3: Entry 22 of 23 File: USPT Aug 13, 1991

US-PAT-NO: 5039421
DOCUMENT-IDENTIFIER: US 5039421 A

TITLE: Solvent stable membranes

DATE-ISSUED: August 13, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Perry; Mordechai	Petach Tikva			IL
Nemas; Mara	Neve Monosson			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.43, 210/654

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw Desc | Image

☐ 23. Document ID: US 5032282 A

L3: Entry 23 of 23 File: USPT Jul 16, 1991

US-PAT-NO: 5032282
DOCUMENT-IDENTIFIER: US 5032282 A

TITLE: Solvent-stable semipermeable composite membranes

DATE-ISSUED: July 16, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.23, 210/500.43, 210/654

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | KWIC | Draw. Desc | Image

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chitosan can be an excellent candidate for filtration membranes. Moreover, since chitosan has a positive charge due to the presence of --NH.sub.2 groups, it can be used to selectively adsorb malignant leukemia cells which carry a higher negative charge on their surface than normal cells. Since chitin contains N-acetyl-D-glucosamine units in its structure which can bind certain molecules, it can be employed for affinity purification without further chemical modification. Other advantages of chitosan and chitin are that they are easily available and inexpensive. Moreover, chitin and crosslinked chitosan are insoluble in both acidic and alkaline media making them suitable as filtration membranes.

Brief Summary Text (8):
In addition to affinity filtration, other uses of chitosan membranes include reverse osmosis (Yang and Zall, 1984 J. Food Sci., vol 49:91-93), pervaporation (Tsugita et al., U.S. Pat. No. 4,983,304; Zeng et al., 1993 Membr. Sci. Technol., vol 13:29-32; Goto et al., 1994 Sep. Sci. Technol. vol 29:1915-23), ultrafiltration (Beppu et al., 1993 Kobunshi Ronbunshu vol 50:35-40), and affinity filtration (Zeng and Ruckenstein 1996 J. Membr. Sci. vol 117:271-278). U.S. Pat. No. 5,116,747 to Moo-Young et al. describes the use of a semi-permeable membrane, formed by chitosan and a water soluble polymer, for immobilization of biologically active material. U.S. Pat. No. 5,006,255 to Uragami describes a selective permeable membrane prepared by cross-linking of chitosan by aldehyde, and used for separation of water-alcohol solution.

Brief Summary Text (9):
Currently, there is no suitable method available for the preparation of microporous or macroporous chitosan membranes wherein the size of the pores can be controlled. The most common method to prepare microporous chitosan membranes is the phase-inversion process, using a large molecular weight organic compound as a porogen. The process involves three steps: (1) casting of a solution of the membrane containing a porogen and partial evaporation of the solvent; (2) sol-gel transformation and generation of pores via the addition of a solvent for the porogen; and (3) heat treatment for stabilizing the pore structure and improving the mechanical properties. This method requires rigorous control of various parameters, particularly the kind and amount of porogen and evaporation conditions (time, humidity and temperature). Generally, the porogens employed in the phase-inversion methods for preparing hydrophobic membranes were organic compounds of low molecular weight such as acetone, dimethyl formamide, dimethyl sulfoxide, benzene, etc. To obtain large pores in chitosan membranes, the relatively large molecule of poly(ethylene glycol), molecular weight 35,000, was used as porogen (Zeng and Ruckenstein, 1996 J. Membr. Sci. vol 117:271-278). Although relatively high permeability membranes were obtained, their mechanical properties were not satisfactory, and they had to be placed on another support.

Brief Summary Text (10):
So far, microporous or macroporous chitin membranes have not been available, primarily because no suitable solvent and porogen could be found. A few solvents, such as the mixtures trichloroacetic acid-chloral hydrate-dichloromethane (Brine and Austin, 1975 ACS Symposium Series, Church T. D., Eds., American Chemical Soc., vol 18, p505), dimethylacetamide (DMAc)-LiCl (Rutherford and Austin, 1977 Proc. of the First International Conf. on Chitin and Chitosan, Muzzaralli, R. A. A., Priser, E. R., Eds., MIT Sea Grant Program, Cambridge), and N-methyl-2-pyrrolidone-DMAc-LiCl (Uragami et al., 1981 Polym., vol 30:1155-1156) have been tried. However, it was either almost impossible to completely dissolve chitin in these solvents, or required a long time, followed frequently by degradation.

Brief Summary Text (12):
An object of the present invention is to provide a macroporous or microporous filtration membrane for affinity purification of macromolecules, wherein the matrix comprises chitosan or its acetylated form, chitin, and the size of the pores can be controlled.

Brief Summary Text (13):
Another object of the present invention is to provide a method for the preparation of chitosan or chitin membranes wherein the matrix of the membrane is formed around a porogen of desired size. The porogen particles are then dissolved to form the

membrane pores.

Drawing Description Text (2):
FIG. 1a is a scanning electron micrograph showing the morphology of a macroporous chitosan membrane, wherein the weight ratio of silica particles to chitosan is 4:1 and the size of silica particles is 5 .mu.m.

Drawing Description Text (3):
FIG. 1b is a scanning electron micrograph showing the morphology of a macroporous chitosan membranes, wherein the size of silica particles is 10 .mu.m and the weight ratio of silica to chitosan is 4:1.

Drawing Description Text (4):
FIG. 1c is a scanning electron micrograph showing the morphology of a macroporous chitosan membranes, wherein the size of silica particles is 15-40 .mu.m and the weight ratio of silica to chitosan is 4:1.

Drawing Description Text (5):
FIG. 2 is a graph illustrating the effect of the size of silica particles on the flow rate through chitosan membranes.

Drawing Description Text (6):
FIG. 3 is a schematic diagram of a cartridge used to house chitosan or chitin membranes for affinity filtration.

Drawing Description Text (7):
FIG. 4 is a graph illustrating the adsorption of lysozyme on the chitin membrane cartridge at 20.degree. C.

Detailed Description Text (2):
"Macroporous" as used herein means pores having a diameter of at least 1.0 .mu.m.

Detailed Description Text (3):
"Microporous" as used herein means pores having a diameter of from 0.1 .mu.m to about 1.0 .mu.m.

Detailed Description Text (4):
"Membrane" as used herein means a membrane wherein the matrix can be of any shape including, but not limited to, flat surfaces and spheres.

Detailed Description Text (5):
The present invention is concerned with a porous affinity membrane wherein the matrix comprises chitosan or chitin, and wherein the pores are created by dissolution of the porogen. The pores can be in the micro--(diameter 0.1 .mu.m to 1.0 .mu.m) or macro--(diameter greater than 1.0 .mu.m) range depending upon the size of the porogen selected for the preparation of the membranes.

Detailed Description Text (6):
To prepare the membrane of the present invention, commercially available chitosan such as, but not limited to, chitosan of molecular weight about 70,000 to about 2,000,000, can be used. While chitosan of any molecular weight can be used, a preferred embodiment has chitosan of molecular weight between about 400,000 to about 2,000,000.

Detailed Description Text (7):
Any porogen in particulate form that is soluble in basic solutions and insoluble in acidic ones can be used. It is preferable to use an inorganic porogen in order to obtain a dried chitosan membrane rather than a chitosan gel membrane. Suitable porogens include, but are not limited to, silica, aluminum silicate and aluminum oxide.

Detailed Description Text (8):
In one illustrative embodiment, the porogen is silica. Since silica is available in several sizes, a wide range of pore size can be achieved. In a more preferred embodiment, the size of silica particles is from about 15 .mu.m to about 40 .mu.m

which results in an average pore size of 19.8 .mu.m for chitosan membranes and 17.9 .mu.m for chitin membranes.

Detailed Description Text (9):
Chitosan membranes are prepared according to the process of the present invention by first preparing a suspension of the porogen particles in an aqueous acidic solution of chitosan. To prepare acidic chitosan solution, organic acids were found to be particularly suitable for this invention. Such acids include, but are not limited to, acetic acid, formic acid, dichloroacetic acid, and trifluoroacetic acid. However, non-organic acids such as, but not limited to, hydrochloric acid, also work well. In a preferred embodiment, the 'acid is acetic acid. The concentration of chitosan in the aqueous acidic solution is inversely related to its molecular weight. For example, for an average molecular weight of 750,000, the appropriate concentration is around 0.5 wt % to 1.5 wt %. For the lower molecular weight of 70,000 the concentration is around 2 wt % to 6 wt percent.

Detailed Description Text (10):
The acidic chitosan solution containing the porogen is shaped into the desired membrane form. For preparing flat sheets, the suspension is cast on a rimmed glass plate and the solvent is allowed to evaporate. For hollow fibers, the pressurized suspension is extruded through a spinneret into a coagulation bath and for preparing beads, the pressurized suspension is dropped through a nozzle into a coagulation bath. The coagulation bath comprises NaOH. In addition, it may also contain ethanol or methanol.

Detailed Description Text (11):
The porogen particles are easily extracted by exposing the shaped membranes to an alkaline solution. It may be desirable to carry out the treatment with the alkaline solution at a higher temperature so as to accelerate dissolution of silica and generate a porous membrane. In addition, heat treatment also improves the mechanical properties of the membrane. The time of exposure to the alkaline solution is dependent upon the temperature. For example, at 80.degree. C., a 2 hour treatment was found to be adequate, while at room temperature, a 24 hour treatment was necessary. Chitosan is insoluble in alkaline solution and hence is unaffected by this treatment.

Detailed Description Text (12):
Following dissolution of the porogen, the alkaline solution is removed by washing with water and then the membrane is either allowed to dry or can be stored in methanol or ethanol. In a preferred embodiment, the to membrane is treated, before drying, with a plasticizer, which acts as a softening agent. This reduces the shrinkage during drying but has only a minimal effect on the flow rate through the membrane. Such plasticizers include, but are not limited to glycerol, ethylene glycol, propylene glycol, diethylene glycol, triethylene glycol and trimethylene glycol. Glycerol is a particularly suitable plasticizer.

Detailed Description Text (14):
The number of pores in a membrane can be controlled by changing the weight ratio of silica to chitosan. For example, increase of this ratio from 1:1 to 2:1 rapidly increases the flow rate of pure water through the membrane prepared with silica particles in the range 15 .mu.m to 40 .mu.m.

Detailed Description Text (15):
The flow rate increases, however, only slightly for ratios larger than 2:1, because the resultant membrane is more easily compressed at higher pressure drops. Membranes prepared with silica particles of other sizes also display a similar behavior.

Detailed Description Text (16):
In affinity separation processes, the proteins or enzymes adsorbed are often eluted at low pH. To prevent the dissolution of the membranes in acidic solutions, the membrane is treated with a cross-linking agent. Any cross-linking agent that reacts with OH or NH.sub.2 can be used. Such cross-linkers include, but are not limited to, glutaraldehyde, hexamethylene diisocyanate, epichlorohydrin, and ethylene glycol diglycidyl ether. However, cross-linking decreases the number of functional groups, and consequently the potential ligand density. The chitosan molecule contains two

functional groups, OH and NH.sub.2, with the latter being more active than the former. In one preferred embodiment, to maintain the number of amine groups, epichlorohydrin is used since, under basic conditions, it reacts only with the OH groups.

Detailed Description Text (17):
Chitin microporous or macroporous membranes are obtained via acetylation of the corresponding chitosan membranes with acetic anhydride in methanol. The N-acetylated chitosan (chitin) membrane has a stronger chemical resistance than chitosan membrane, being insoluble in 5 vol % aqueous solution of acetic acid (pH 2.5) and in 5 wt % aqueous NaOH solution. This increased chemical resistance is most likely due to the presence of COCH.sub.3 group, which decreases the elongation upon increasing the extent of crystallinity.

Detailed Description Text (18):
The method of the present invention can also be employed to prepare composite membranes, in which chitosan is blended with synthetic polymers such as, but not limited to, polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), polyethylene oxide (PEO), poly amides, polyacrylamides, polymethacrylate and polyhydroxyethyl methacrylate, or natural polymers such as, but not limited to, gelatin, collagen, dextran, agarose, silk, cellulose and cellulose derivatives. Such combinations would be useful in improving the properties of polymer membranes, such as blood compatibility, mechanical properties and biodegradability.

Detailed Description Text (19):
The present invention is not limited to flat sheets, but can be used in other forms including, but not limited to, hollow fibers and beads. Flat membranes can be housed in a support assembly. In a preferred embodiment, the support assembly is a plate type filtration cartridge wherein, multiple membranes can be stacked. Stacking of multiple membranes increases the adsorptive capacity of the membranes.

Detailed Description Text (20):
In contrast to most commercial membranes, the membranes of the present invention contain a large number of active groups (--OH and/or NH.sub.2). Therefore the membranes of the present invention can be used, without any further amplification of the number of active groups, in various applications such as affinity membranes. Since chitin membranes contain N-acetyl-D-glucosamine units in its structure, it can be used to bind macromolecules that have an affinity for this group. Such macromolecules include, but are not limited to, lysozyme and wheat germ agglutinin.

Detailed Description Text (21):
Chitosan membranes can bind molecules that have an affinity for glucosamine. Such molecules include, but are not limited to, protein A and Cibacron.TM. Blue F3GA dye. Other potential uses for macroporous and microporous chitosan and chitin membranes include, but are not limited to, agents for wound dressing, hemostatic bandages, metal chelating agents, enzyme carriers, agents for cell immobilization, and blood filters to remove selected cells.

Detailed Description Text (24):
Preparation of Chitosan Macroporous Membranes

Detailed Description Text (25):
Illustrated in this embodiment is the preparation of chitosan membranes. A solution of chitosan was first obtained by dissolving 1 g of chitosan in 100 ml of 1 vol % aqueous acetic acid solution containing 10 wt % glycerol. To this solution, the desired size and amount of silica particles were added, followed by vigorous stirring in order to disperse them uniformly. Then the solution was poured onto a rimmed glass plate and the liquid was allowed to evaporate. The dried membrane was immersed in a 5 wt % aqueous NaOH solution and kept for 2 hours at 80.degree. C. Finally, the porous membrane was washed with distilled water to remove the remaining NaOH.

Detailed Description Text (27):
Cross-linking of Microporous or Macroporous Chitosan Membranes

Detailed Description Text (28):
While insoluble in alkaline solutions, the chitosan membrane is soluble in dilute acetic acid solutions. To prevent the dissolution of chitosan membrane under acidic conditions, which are necessary to elute many biomolecules, the chitosan membrane must be cross-linked. Cross-linking of the chitosan membrane was carried out under mild conditions using epichlorohydrin as the cross-linker. The chitosan membranes were immersed in 1.times.10.sup.-2 M epichlorohydrin solution, containing 0.067M NaOH (pH 10) for 2 hours at 50.degree. C. Then the membranes were taken out of the solution and rinsed with distilled water until neutral conditions. Table 1 shows the effect of cross-linking of chitosan membranes.

Detailed Description Text (29):
In order to prevent its shrinkage during drying, the membrane was immersed in a 20 vol % aqueous glycerol solution for 30 min and, after removing the excess glycerol solution, placed on a glass plate and allowed to dry. Thus, a strong and flexible membrane that had not undergone shrinkage, was obtained.

Detailed Description Text (31):
Conversion of Macroporous Chitosan Membranes to Macroporous Chitin Membranes

Detailed Description Text (32):
To prepare chitin membranes, the corresponding chitosan membranes were acetylated after removal of the alkaline solution used to dissolve the porogen particles. The acetylation of chitosan membranes to chitin was carried out via its immersion into a stirred solution of 100 ml methanol containing 5 ml of acetic anhydride for 1 hour at 50.degree. C. The membranes were then removed from the solution and washed successively with methanol and distilled water, followed by treatment of the membrane with 5 wt % aqueous NaOH solution overnight to remove the CH.sub.2 OH acetylated groups. Finally, a white macroporous chitin membrane was obtained after washing with distilled water until neutral conditions. Table 2 presents a comparison of the chemical resistance and mechanical properties, and Table 3 presents a comparison of the physical properties of chitin and chitosan membranes. The mechanical properties of the chitosan and chitin membranes were determined at 20.degree. C. using an Instron.TM. universal testing instrument(Model 1000). The gauge length was 20 mm and the extension rate 10 mm/min. The specific adsorption areas of chitosan and chitin macroporous membranes were determined by the BET (Brunauer-Emmett-Teller) method using a Micromeritics.TM. ASAP 2000 instrument. The porosities of the chitosan and chitin membranes were obtained by determining their swelling in water and using the following expression:

Detailed Description Text (33):
where W.sub.1 and W.sub.2 are the weights of the membranes in the wet and dry states, respectively, d.sub.water is the density of pure water at 20.degree. C., and V is the volume of the membrane in the wet state.

Detailed Description Text (35):
Morphology of Chitosan and Chitin Membranes:

Detailed Description Text (36):
Scanning electron microscopy was employed to investigate the morphology of the chitosan and chitin membranes. The specimen were prepared as follows: the wet membrane was wiped with a filter paper to remove the excess water present on the surface of the membrane, then framed on a petri dish to prevent shrinkage along the surface, and allowed to dry. The membranes were fractured under liquid nitrogen and the fractured surfaces were coated with a thin layer of carbon before scanning. FIGS. 1(a), 1(b) and 1(c) show that the pores are distributed uniformly indicating that the silica particles were dispersed uniformly.

Detailed Description Text (38):
Effect of Size of Silica Particles on the Morphology and Flow Rate through Chitosan Membranes:

Detailed Description Text (39):
To determine the effect of the size of silica particles on the morphology of the membrane and on the rate of flow of fluids through the membrane, three kinds of

silica particles, size between 15 and 40 .mu.m, average size of 10 .mu.m, and average size of 5 .mu.m, were selected for illustration. The morphologies of the chitosan membranes can be seen from the electron micrographs of FIG. 1a (5 .mu.m), FIG. 1b (10 .mu.m), and FIG. 1c (15-40 .mu.m), which show three-dimensional networks with high porosities and uniformly distributed pores. A comparison of the flow rates through these membranes is presented in FIG. 2. The flow rate for membranes prepared with silica particles of sizes 15-40 .mu.m (curve 1) and 10 .mu.m (curve 2) was higher than that for membranes prepared with silica particles of size 5 .mu.m (curve 3). The larger silica particles provided the larger pore sizes and, hence, the larger flow rates of pure water through the membrane. The average pore size of membranes prepared with a weight ratio of silica to chitosan of 8:1 and with silica particles of sizes 15-40 .mu.m, 10 .mu.m and 5 .mu.m membranes was 19.5 .mu.m, 6.6 .mu.m, and 2.5 .mu.m respectively. These results indicate that the size of the pores can be controlled by selecting silica particles of appropriate size. In addition, the electron micrographs indicate that the pores are uniformly distributed.

Detailed Description Text (41):
Preparation of Composite Membranes containing Chitosan and Synthetic Polymers

Detailed Description Text (42):
In one embodiment of the invention, chitosan is blended with synthetic polymers to make composite membranes. Chitosan and PEO were dissolved individually in 1 vol % aqueous acetic acid solution containing 10 wt % glycerol. Then the two solutions were mixed in various proportions. The silica particles (15-40 .mu.m) were suspended via stirring in the mixture; this was followed by casting the suspension on a rimmed glass plate. After drying at room temperature, the dried membrane was immersed in 5% NaOH solution at 80.degree. C. for 2 hours, followed by washing with distilled water. This produced a macroporous chitosan-poly(ethylene oxide) blend membrane.

Detailed Description Text (43):
To prepare a chitosan-polyvinyl alcohol blend membrane, the casting solution was prepared by mixing a 2 wt % polyvinyl alcohol (PVA) aqueous solution with a 1 wt % chitosan aqueous acetic acid solution containing 10 wt % glycerol and silica particles (15-40 .mu.m). Dissolution of silica particles was carried out by immersion in 5% NaOH at 60.degree. C. for 5 hours.

Detailed Description Text (45):
Preparation of Composite Membranes containing Chitosan and Natural Polymers

Detailed Description Text (46):
In one embodiment of the invention, composite membranes containing chitosan and collagen or chitosan and gelatin were prepared. One wt % solutions of chitosan and collagen or gelatin were prepared by dissolving them individually in a 2 vol % aqueous acetic acid solution. The individual solutions were then mixed in a 1:1 volume ratio. Silica particles (15-40 .mu.m) were added with vigorous mixing, followed by casting the suspension on a rimmed glass plate and drying at room temperature. The dried membrane was immersed in 5% NaOH solution at 60.degree. C. for 5 hours, followed by washing with distilled water.

Detailed Description Text (48):
Preparation of membrane cartridge

Detailed Description Text (49):
In one embodiment of this invention, the membrane is housed in a plate type filtration cartridge. While any cartridge known to those skilled in the art can be used, an example of such a cartridge is presented here. A schematic diagram of the cartridge is presented in FIG. 3. Two porous sintered plates (3 mm thick) were employed as distributors in the cartridge. In addition, multi-inlet ports (4 inlets) and a bubble relief valve were employed in order to achieve better sample distribution. The cartridge has a hollow bottom portion 5, the bottom of which houses a flow collector 8 which leads to an outlet port 6. The stacked chitosan membranes 7 are placed on the flow collector. The top cover of the cartridge 4 has a flow distributor 3 which, together with the flow collector 8, sandwiches the chitosan membranes 7. The flow distributor 3 in the cover 4 has three inlet ports 2 to feed the fluid to the membranes. The purpose of the flow distributor is to feed

the fluid uniformly to the membranes. The cover houses an "O"-ring seal 1, which seals the chitosan membranes from the outside.

Detailed Description Text (52):
In one embodiment of this invention, the membrane is in the form of a hollow fiber. For preparing hollow fibers, an acidic chitosan solution containing silica particles of desired size is placed into a cylinder and extruded with a piston through a spinneret into a coagulation bath (aqueous 5% wt NaOH, which may contain ethanol or methanol). While not intending to be bound by any particular theory, it is believed that this solidifies the fiber by deprotonating the amine group. This is followed by drawing the fiber through a washing bath (deionized water) to remove the sodium hydroxide, and then through an acetone bath to dehydrate the fiber. Glycerine may be added into the spin dope, the coagulating solution and washing bath to prevent rupture during drying. Subsequently the fiber is immersed in a NaOH solution at 80.degree. C. to dissolve the silica particles and to generate the porous fiber. A hollow fiber spinneret can be employed to prepare chitosan hollow fiber. Treatment with a cross-linker, as in Example 2, is needed to stabilize the membranes. Micro- or macroporous chitin fibers or hollow membranes can be prepared by acetylating the chitosan fiber or hollow fiber with acetic anhydride.

Detailed Description Text (58):
Use of the Membrane:

Detailed Description Text (59):
In one embodiment of the invention, the membrane is used for affinity purification of lysozyme which has a known affinity for the D-glucosamine moieties of chitin. A 1 mg/ml solution of lysozyme in 0.1 M phosphate buffer (pH 8.0) containing 1 M NaCl was prepared and loaded at a flow rate of 1, 5, or 15 ml/min into the chitin cartridge of Example 8. The ratio of the concentration of lysozyme in the effluent (C) and the initial concentration of lysozyme (C.sub.o) is plotted as a function of time in FIG. 4. The time required to achieve saturation was about 20 min for 15 ml/min (c; curve 6), about 30 min for 5 ml/min (b; curve 5) and more than 70 min for 1 ml/min (a; curve 4). The adsorption was followed by washing with phosphate buffer for 10 min at a flow rate of 15 ml/min, and by elution with 0.1 M acetic acid solution at 1, 5, and 15 ml/min, until no protein was detected in the effluent. The effluent was collected and the concentration determined spectrophotometrically. The elution profile is presented in FIG. 5. About 40, 10 and 5 minutes were needed for the elution flows of 1 ml/min (a; curve 7), 5 ml/min (b; curve 8), and 15 ml/min (c; curve 9) respectively to remove 95% of the strongly bound protein.

Detailed Description Text (61):
Use of the Membrane to Purify Lysozyme from Egg White

Detailed Description Text (62):
In one embodiment of the invention, the membrane was used for separation of lysozyme from egg white. Hen egg white was first separated from fresh eggs. Then 10 ml of homogenized egg white was diluted with 90 ml of 0.1 M phosphate solution (pH 8.0) containing 1 M NaCl, followed by filtration and centrifugation at 100 g for 20 min. Finally, 65 ml of supernatant was pumped through the chitin cartridge of Example 8, at 1 ml/min, followed by 10 min washing at 15 ml/min and about 15 min elution at 5 ml/min. The purity of the lysozyme was examined by High Performance Liquid Chromatography (HPLC) using a wide-pore CBx HPLC column, 5 .mu.m, 7.75 mm.times.100 mm. The flow rate was 1 ml/min, the mobile phase A binding buffer was 25 mM MES ((2-N-morpholino) ethanesulfonic acid), pH 5.6 and the mobile phase B eluting buffer was 1 M NaOAc, pH 7.0. The sample size was 100 .mu.l. The various profiles in FIG. 6 represent pure ovalbumin (a; curve 10), pure lysozyme (b; curve 11) and lysozyme from egg white (c; curve 12). The purity of lysozyme from egg white was estimated to be higher than 98% and its specific activity was 54,003 units/mg protein.

Detailed Description Paragraph Table (1):
TABLE 1 _____ in 5 vol % chitosan aqueous acetic acid flux (ml/min/cm .sup.2) membrane soln, pH 2.5 at pressure drop of 3/5 psi _____ before cross-linking soluble 18.1/27.4 after cross-linking insoluble 17.3/26.0 _____

Detailed Description Paragraph Table (2):
TABLE 2 _____ chem resistance in mechanical properties (dry/wet) elongation 5 wt % 5 vol % tensile strength, at membrane NaOH solution HOAc solution MPa break, _____ % chitosan insoluble soluble 7.37/0.90 6.1/102.2 chitin insoluble insoluble 9.23/1.09 4.6/29.3

_____

Detailed Description Paragraph Table (3):
TABLE 3 _____ flux (ml/min/ cm.sup.2) at spec average pressure thickness, porosity, adsorption pore size drop of membrane .mu.m % area m.sup.2 /g .mu.m 3/5 psi _____ chitosan 119 75.2 1.8 19.8 17.6/30.8 chitin 132 62.2 1.6 17.9 15.0/28.9

_____

Other Reference Publication (1):
T. Uragami, Y. Ohsumi and M. Sugihara, (1981), Studies on synthesis and permeability of special polymer membranes; Polymer, vol. 22, pp. 1155-1156.

Other Reference Publication (2):
X. Zeng and E. Ruckenstein, (1996), Supported chitosan-dye affinity membranes and their protein absorption; J. Membrane Sci., vol. 117, pp. 271-178.

Other Reference Publication (3):
T. Yang and R.R. Zall, (1984), Chitosan Membranes for Reverse Osmosis Application; J. Food Science vol. 49, pp. 91-93.

Other Reference Publication (4):
M. Goto, A.A. Shiosaki and T. Hirose, (1994), Separation of Water/Ethanol Vapor Mixtures through chitosan and cross-linked chitosan membranes; Separation Science and Tech., vol. 29, pp. 1915-1923.

Other Reference Publication (5):
M. Beppu, T. Baba and C. Kamizawa, (1993), Effect of organic solvents as gelating agents on performance of chitosan membranes for ultrafiltration; Kobunshi Ronbunshu, vol. 50, pp. 35-40.

Other Reference Publication (6):
X. Zheng, C. Mingde, Z. Xurong and S. Jun, (1993), Pervaporation separation of ethanol-water mixture using crosslinked chitosan/PAN membrane; Membrane Sci. & Technol., vol. 13, pp. 29-32.

CLAIMS:

1. A membrane selected from the group consisting of a microporous and macroporous membrane, wherein said membrane is composed of a matrix and a set or pores, wherein said matrix consists essentially of chitosan, and wherein said pores are formed by dissolution of a particulate porogen, wherein said pores are uniformly distributed and include a three-dimensional structure.

2. A membrane selected from the group consisting of a microporous membrane and macroporous membrane wherein said membrane is composed of a matrix and a set or pores, wherein said matrix comprises chitosan and at least one polymer selected from the group consisting of polyethylene oxide, polyvinyl alcohol, collagen and gelation, and wherein said pores are uniformly distributed and have a three dimensional structure.

3. The membrane of claim 2, wherein said polymer is polyethylene oxide.

4. The membrane of claim 2, wherein said polymer is polyvinylalcohol.

5. The membrane of claim 2, wherein said polymer is collagen.

6. The membrane of claim 2, wherein said polymer is gelatin.

7. The membrane according to claim 1, wherein said porogen comprises silica

particles.

8. The membrane according to claim 7, wherein the membrane is a macroporous membrane, and wherein said silica particles have a diameter of between about 15 .mu.m to about 40 .mu.m.

9. The membrane according to claim, 1 wherein chitosan has a molecular weight of between about 400,000 to about 2,000,000.

10. The membrane according to claim 1 wherein chitosan is N-acetylated.

11. A method for the preparation of a membrane according to claim 10, comprising the steps of:

a. suspending porogen particles in an acidic solution comprising chitosan

b. shaping the suspension into a membrane;

c. extracting the porogen by contacting the membrane with an aqueous alkaline solution;

d. removing the alkaline solution; and

e. converting chitosan to chitin by treating the membrane with acetic anhydride.

13. The method of claim 12, wherein the silica particles have a diameter of between about 15 .mu.m to about 40 .mu.m, wherein chitosan has a molecular weight of between about 400,000 to about 2,000,000, and wherein the porogen is extracted by heating in an alkaline solution comprising sodium hydroxide.

18. The method according to claim 11, further comprising the step of treating the membrane with a plasticizer to soften the membrane, after step (e).

22. The membrane according to claim 1, wherein the membrane is in the form of a flat sheet.

23. The membrane according to claim 1, wherein the membrane is in the form of a hollow tube or fiber membrane.

24. The membrane according to claim 1, wherein the membrane is in the form of a bead.

25. A method for the preparation of a membrane according to claim 1, comprising the steps of:

a. suspending porogen particles in an acidic solution comprising chitosan;

b. shaping the suspension into a membrane;

c. extracting the porogen by contacting the membrane with an aqueous alkaline solution;

d. removing the alkaline solution; and

e. treating the membrane with a cross-linker.

27. The method of claim 26, wherein the silica particles have a diameter of between about 15 .mu.m to about 40 .mu.m, wherein chitosan has a molecular weight of between about 400,000 to about 2,000,000, wherein the porogen is extracted by heating in an alkaline solution comprising sodium hydroxide, and wherein the cross-linker is epichlorohydrin.

34. The method according to claim 25, further comprising the step of treating the membrane with a plasticizer to soften the membrane, after step (e).

38. A method for the purification of molecules, wherein the molecules have a known affinity for the membrane according to claim 1, comprising the steps of:

a. contacting the molecules in solution with said membrane;

b. washing the membrane to remove unbound molecules; and

c. eluting bound molecules from said membrane with an acidic solution.

39. The method of claim 38, wherein the membrane is housed in a support assembly.

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END OF SEARCH HISTORY